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The crystal structure of 3,10-bis(4-methoxyphenyl)-6,12-dibenzyl-2,9-acetyl-6,12-diazapentacyclo[6.3.1.0_{2,7}.0_{4,11}.0_{5,9}]dodecane – acetone (1/1), C₄₅H₄₈N₂O₅

Table 1: Data collection and handling.

Crystal:	Colourless monoclinic
Size:	0.36 × 0.26 × 0.14 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.82 cm ^{−1}
Diffractometer, scan mode:	Rigaku XtaLAB P200, φ and ω
θ max, completeness:	52.7°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	38645, 7481, 0.0840
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 5642
$N(\text{param})_{\text{refined}}$:	476
Programs:	Rigaku [1], Olex2 [2], SHELX [3, 4]

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Abstract

C₄₅H₄₈N₂O₅, monoclinic, $P2_1/c$ (no. 14), $a = 21.3971(8)$ Å, $b = 15.2249(6)$ Å, $c = 11.2610(5)$ Å, $\beta = 92.125(4)^\circ$, $V = 3666.0(3)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0519$, $wR_{\text{ref}}(F^2) = 0.1308$, $T = 128.15$ K.

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The title crystal structure has been shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

The title compound was synthesized by [2 + 2] photocycloaddition reaction. 3-Acetyl-*N*-benzyl-4-methoxyphenyl-1,4-dihydropyridin was selected as the substrate under irradiation of 410 nm UV-light in methanol/THF solvent. After 7 days, the product was purified by silica gel (P:E = 50:1, V/V). After

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	3572.3(6)	5027.7(9)	9236.9(11)	28.1(3)
O2	611.2(6)	4540.5(9)	7973.4(12)	33.7(3)
O3	642.3(6)	8319.1(9)	4190.9(12)	32.0(3)
O4	4134.7(6)	563.9(8)	7695.5(12)	31.3(3)
N1	2193.4(6)	5805.6(9)	7380.5(13)	19.2(3)
N2	2201.0(6)	3491.3(9)	7862.7(12)	19.0(3)
C1	1881.5(8)	5029.8(11)	7834.7(15)	19.0(4)
C2	2416.7(8)	4360.6(11)	8231.4(15)	18.9(4)
C3	2980.2(8)	4636.3(11)	7461.3(15)	19.0(4)
C4	2678.6(8)	5409.4(11)	6680.4(15)	19.4(4)
C5	1919.2(8)	3677.1(11)	6689.8(15)	19.2(4)
C6	1523.1(8)	4550.0(11)	6793.4(15)	19.2(4)
C7	1637.5(8)	5000.8(11)	5596.7(15)	19.6(4)
C8	2355.8(8)	4920.0(11)	5594.9(15)	19.7(4)
C9	2461.9(8)	3939.4(11)	5852.5(15)	19.7(4)
C10	3097.9(8)	3919.0(11)	6523.5(15)	20.0(4)
C11	3562.2(8)	4932.1(11)	8164.3(17)	22.4(4)
C12	4143.4(9)	5108.7(15)	7481.9(19)	35.1(5)
C13	834.7(8)	4377.5(12)	7023.1(17)	23.0(4)
C14	434.3(9)	3986.7(15)	6040.9(19)	37.5(5)
C15	2409.8(8)	6390.3(11)	8339.1(16)	21.8(4)
C16	2655.9(8)	7265.8(12)	7926.7(16)	22.0(4)
C17	2443.3(9)	7658.2(12)	6880.4(17)	27.0(4)
C18	2633.3(10)	8506.6(13)	6604.7(19)	33.6(5)
C19	3041.2(10)	8958.4(14)	7364(2)	37.3(5)
C20	3255.5(9)	8567.6(14)	8406(2)	36.7(5)
C21	3063.8(9)	7726.4(13)	8686.2(19)	30.3(4)
C22	1784.6(8)	3069.6(11)	8701.2(16)	22.0(4)
C23	1559.3(8)	2198.4(12)	8214.9(16)	22.7(4)

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Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C24	927.5(9)	1989.8(14)	8171.5(19)	33.8(5)
C25	715.7(11)	1199.6(15)	7703(2)	43.7(6)
C26	1135.9(12)	611.8(15)	7263(2)	46.3(6)
C27	1769.5(11)	809.1(14)	7293.5(19)	38.7(5)
C28	1980.9(9)	1595.4(12)	7771.9(17)	28.3(4)
C29	1355.2(7)	5892.1(11)	5303.2(15)	19.3(4)
C30	986.4(8)	6369.4(12)	6056.2(16)	21.6(4)
C31	735.2(8)	7181.5(12)	5724.5(16)	23.2(4)
C32	853.3(8)	7524.1(12)	4615.7(16)	22.3(4)
C33	1216.6(8)	7050.2(12)	3844.3(16)	23.6(4)
C34	1463.1(8)	6249.0(12)	4190.4(16)	22.1(4)
C35	268.5(11)	8829.6(14)	4950(2)	42.7(6)
C36	3354.8(8)	3031.1(11)	6912.0(16)	19.8(4)
C37	3331.1(8)	2327.9(12)	6112.2(17)	24.3(4)
C38	3584.8(8)	1517.1(12)	6395.8(17)	26.0(4)
C39	3881.3(8)	1385.7(12)	7499.6(17)	23.8(4)
C40	3907.5(8)	2064.8(12)	8316.3(17)	25.1(4)
C41	3646.2(8)	2875.6(12)	8010.5(16)	23.4(4)
C42	4401.2(9)	388.8(13)	8844.2(18)	32.0(5)
O5	4397.0(9)	6885.9(14)	5636(2)	78.7(7)
C43	4811.9(13)	7562(2)	3981(3)	66.0(8)
C44	4556.9(10)	7560.4(18)	5177(2)	47.4(6)
C45	4515.7(19)	8419(2)	5783(3)	93.1(12)

concentration in vacuum, freeze-drying, a colorless powder was obtained. The powder was recrystallized from acetone. Two days later the crystals of the title compound were harvested.

Experimental details

All hydrogens were placed in idealized positions and added using the riding models implemented in the SHELX system. Using Olex2 [2], the structure was solved with the ShelXT [3] structure solution program using intrinsic phasing and refined with the ShelXL [4] refinement package using least squares minimisation.

Discussion

Organic photochemical reactions have advantages of easy operation methods, high efficiency and more environmental-friendly conditions, controllable cost and so on. The products of the photochemical reaction found may have many kinds of pharmacological activities [5, 6]. Despite all efforts, accessibility of cage compounds analogous to cubane is limited [7].

Only a small number of 6,12-diazatetrakisomocubanes have been reported so far [8].

The structure of title compound was determined by X-ray diffraction analysis. The crystal structure revealed that the two piperidine rings are in boat conformation, the apexes (prows) being the N1, C7 and N2, C10 atoms, and the five-membered rings adopt an envelope form conformation in the compound (see the figure). The compound is a cage structure, which belongs to the class of 6,12-diazatetrakisomocubanes [7–9]. The feature of torsion angle are C1–N1–C15–C16: –171.85°, C4–N1–C15–C16: 72.46°, C2–N2–C22–C23: 176.94°, C5–N2–C22–C23: 60.29°.

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